Verband der
Mineralfarben-
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VdMi statement on Requirements of the 15th Amendment of the Plastics Regulation (EU) No 10/2011 and the associated difficulties in practical implementation for the manufacturers of pigments and masterbatches

The 15th Amendment of the Plastics Regulation, which was published in the Official Journal on 3 September 2020 as Regulation (EU) 2020/1245 and entered into force on 23 September 2020, includes among others the following changes:

- *The adaptation of the specific migration limits SML (ANNEX II (1)) for certain elements/heavy metals, essentially lowering.*
- *Innovations in the analysis of chromium, in particular the differentiation of chromium VI/III*
- *New passage in the specific migration limits SML for PAA (Annex II (2))*
- *Amendment in Annex IV in the declaration of compliance (point 6), mandatory quantity information on SML-relevant substances from Annex II for intermediates*
- *Information on substances with genotoxicity*

Summary:

In 2019 we took the opportunity to communicate our concerns to the legislator (EU Commission, BMEL). These industry submissions, among others from VdMi and Eurocolour, with which we explicitly drew attention to the difficulties as to analytics and communication in the supply chain, were not taken into account; this was subsequently justified with the high workload of the Commission.

The pigment and masterbatch manufacturers, as suppliers (raw material and intermediate product) in the value chain, can only make a reliable statement about migration from the end product with great difficulty.

The obligatory quantity declaration of all SML-relevant substances according to Annex II and exact assessment (for migration in the end product), which are contained or may be contained in a raw material or intermediate or are definitely not contained, is not practicable and in our view not adequate information. This affects the manufacturers of masterbatches as typical intermediates and the manufacturers of pigments and fillers as raw materials. This new requirement in Annex IV point 6 is to be implemented within the plastics supply chain.

Recital 27 provides the following justification for the quantity declaration:

(27) The new or updated restrictions on substances in Annex II require clear communication in the supply chain to ensure that adequate information on the presence of these substances is available to business operators which use products from intermediate stages in the supply chain or final articles or materials in which these substances may be contained. When such information is not provided they cannot be certain on the presence and amount of these substances and they would need to test more frequently than would be needed if that information was provided. However, if the presence and amount of these substances is known to these business operators, in many cases simple calculation techniques can suffice to establish whether a limit could be exceeded, and analytical testing would not be required at all. Moreover, communication of the amounts of substances is also required to communicate on the presence of these substances to later stages of the supply chain.

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Therefore, it is appropriate to amend point 6 of Annex IV to the Regulation to clarify that the amount of substances subject to limits under Annex II should be included in the declaration of compliance.

At present, the newly required data are not available, which leads to further enquiries and considerable analysis effort. The masterbatch and pigment manufacturers comment jointly on the analytical challenges and practical problems as well as difficulties in implementation and they also point out possible solutions.

Inorganic pigments

Numerous inorganic pigments have been used for decades in masterbatches for colouring plastics. These include complex inorganic coloured pigments, iron oxides and titanium dioxide. Many of these substances are natural components of the biosphere.

Pigments are insoluble in the application medium. Any solubility would be visible and trigger complaints from users.

In addition, studies show that in the case of pigments and fillers bound in a plastic matrix, migration of (nano-)particles and thus transfer to foodstuffs can be ruled out if the particles are larger than 2-3 nm.

This has been proven in migration studies by the Fraunhofer Institute in Freising. The following can be found in the VdMi paper “Facts on the Nano Discussion in the Pigment and Filler Industry”.

The Fraunhofer study on the migration of nanoparticles:

Pigments and fillers are frequently used in plastics, coatings and printing inks that come into contact with food. Here, it must be ensured that there is no migration into the foodstuff. As many pigments and fillers fall under the existing EU Commission Recommendation on the Definition of Nanomaterial, the question is increasingly gaining in importance whether nano particles migrate from plastics or printing inks and, consequently, whether they can migrate into foodstuffs. Based on migration studies from plastics and on theoretical considerations it was possible to demonstrate that a particle migration can be excluded for particles with a size of more than 2 or 3 nm.

Chromium analysis

In Annex II, Remark 3 states the following for the analysis of chromium:

(3) To verify compliance with the Regulation, the detection limit of 0,01 mg/kg shall apply for total chromium. However, if the operator that placed the material on the market can prove on the basis of pre-existing documentary evidence that the presence of hexavalent chromium in the material is excluded because it is not used or formed during the entire production process, a limit for the total chromium of 3,6 mg/kg food shall apply.

Chromium is a common element in the biosphere. There is currently no possibility to analytically exclude the presence of chromium (VI) in very small traces. This applies to almost all chromium-containing pigments. Therefore, the migration value of 0.01 mg/kg on the end product would always apply, which is very low from our point of view. There is no method for the analysis of chromium (VI) in the ppb range that is applicable to insoluble materials.

Pigment and masterbatch manufacturers can only assess with great difficulty, whether the migration value of 0.01 mg/kg can be complied with. A worst-case calculation using the example of chromium shows that this cannot replace the analysis on the end product, the food contact material (FCM).

When adding 5 % of a selected pigment to the masterbatch (intermediate), the worst-case calculation results in values in the FCM that make a migration analysis on the end product, the final FCM, necessary.

Calculation example: Pigment Brown 24, inorganic coloured pigment

- Chromium content 3.6 % = 36 000 ppm
- Soluble chromium-(VI) content = 3 ppm, hydrochloric acid extract 0.1 M according to AP(89)1
- Addition of pigment to intermediate (masterbatch) = 5 %
- Addition of intermediate (masterbatch) into the FCM = 6 %

Worst case calculation of total chromium content:

→ 36 000 ppm x 5 % = 1800 ppm [concentration in intermediate material]
→ 1 800 ppm x 6 % = 108 ppm [concentration in FCM]
→ Migration analysis on FCM necessary

Calculation with soluble chromium-(VI) content:

→ 3 ppm x 5 % = 0.15 ppm [concentration in intermediate material]
→ 0.15 ppm x 6 % = 0.009 ppm [concentration in FCM]
→ < 0.01 ppm (with 100 % migration)

A migration of 100 % is not realistic. In addition, with the analytical value and the resultant content of the migrant in the FCM, further migration calculations are possible. These are described at the end of this statement.

In the mandatory quantity specification required by the legislator, the differentiation between insoluble and soluble components is missing. The specification of a total quantity often leads to extremely high maximum values, as constituent components are also affected. This makes an analysis of the completed FCM mandatory necessary. In our view, this is in clear contradiction to the objective in recital 27, i. e. to reduce analysis at the final product by providing additional information at the beginning of the supply chain.

A common analytical method for pigments in practice is the hydrochloric acid extraction method. The results are also communicated to customers, if required.

Hydrochloric acid 0.1 N or 0.07 N is the simulant in the Toy Safety Directive (stomach simulant), which is based on the assumption (worst case) that a child swallows a building block (oral intake).

The proportion soluble in 0.07 N hydrochloric acid, determined according to DIN 53770, is recorded. The basis is *BfR Recommendation IX for colourants for colouring plastics and other polymers for consumer articles*. The BfR recommendation does not include all elements from Annex II, but in practice almost all of them are determined.

The food simulant „B“ in the Plastics Regulation is 3,0 % w/v acetic acid. However, hydrochloric acid 0.07 / 0.1 N has a greater dissolving power (worst case) than acetic acid and some eco-labels are also based on a hydrochloric acid extract.

Organic pigments

Organic pigments may also contain heavy metals. As a rule, the total metal content can be specified, unless the heavy metal is contained constitutionally.

The Toys Directive also has very low migration limits (SML) for heavy metals, which cannot be achieved by means of state-of-the-art analysis.

For this purpose, there is an ETAD recommendation table for organic pigments, with which confirmation (compliance with the total metal content) is possible by specifying the maximum amount to be added, i. e. without analysis.

This option is limited to organic pigments, it is not applicable for inorganic pigments.

Annex II new passage for primary aromatic amines (PAAs)

Here, too, a worst-case calculation (everything migrates) would lead to an SML limit being exceeded for many pigments. A confirmation via a worst-case calculation is not appropriate for PAAs, because there is no migration study on the end product to determine a factor for the actual migration (x % migrate). Measurements already carried out show that only a relatively small amount migrates to the FCM. Experience also shows that migration is very dependent on the production processes used in the manufacture of the FCM.

A feasible approach would be to state which PAAs are to be expected, together with a quantity range (e. g. 20 / 50 / 100 ppm), a further, more precise specification is not possible. This does not generally avoid the need for a migration analysis on the FCM.

Genotoxic substances

From our point of view, the new requirements from Annex IV point 6 on genotoxic substances cannot be met, because migration can never be completely ruled out.

> *„[…] for which genotoxicity has not been ruled out, and which originate from an intentional use during a manufacturing stage of that intermediate material and which could be present in an amount that foreseeably gives rise to a migration from the final material exceeding 0,00015 mg/kg food or food simulant.“*

Only in rare exceptional cases can genotoxicity be excluded for a product. For example, for large quantities products (pigments), genotoxicity tests were carried out under REACH.

The indication of substances for which genotoxicity cannot be excluded and which can potentially migrate in the quantities mentioned concerns impurities that can arise both from the raw materials used and from degradation reactions during the processing step.

This concerns an evaluation of all NIAS (non-intentionally added substances) in the masterbatch. In order to be able to make a relevant statement on substances and quantities, this could only be assessed with an additional NIAS screening on the masterbatch.

The analytical identification of unknown substances in mixtures is a very demanding challenge, which requires complex analytics. The prediction of migration in the most diverse compositions and application polymers and applications is not possible by means of a theoretical observation.

NIAS can be formed or recorded in the subsequent processing steps.

The analytical effort would double with this approach, as such NIAS screenings have to be carried out again on the final article due to downstream processing.

Calculation / Model calculation

A migration modelling, a model calculation, would be possible for masterbatches based on the analysis of the soluble part of a substance (e. g. hydrochloric acid extraction method).

In the tool *"Note for guidance for petitioners presenting an application for the safety assessment of a substance to be used in food contact materials prior to its authorisation"* from the EFSA (European Food Safety Authority) it is described under point "*3. Calculation of the maximum possible migration"* how the maximum migration can be calculated on the basis of the content of the migrant in the polymer sample.

For this purpose, the content of the migrant in the polymer is determined, e. g. by exhaustive extraction or dissolution of the polymer.

This method has the advantage that the results can be easily extrapolated to any other food contact article made from the same polymer, requiring only one test.

In addition, PlasticsEurope has prepared a *“Risk Assessment of non-listed substances (NLS and non-intentionally added substances (NIAS) under Article 19 of Commission Regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact* *with food"*. Under point 2.2.1.1, a formula is given with which a worst-case calculation of migration is possible.

Furthermore, there is the option of using model calculations to specify a maximum possible amount of masterbatches to be added at which the limit values in the end product will probably not be exceeded.

Migration modelling

By means of a calculation example for chromium (VI), it is to be shown whether, taking into account the communicated solubility in hydrochloric acid and a subsequent migration calculation, the migration content of chromium (VI) in the food is < 0.0012 mg/kg and a health hazard can thus be excluded (recital (19) of Regulation (EU) 2020/1245). When considering the SML of total chromium in the FCM, the SML 3.6 mg/kg should then apply instead of 0.01 mg/kg.

Calculation example: Pigment Brown 24, inorganic coloured pigment (example page 3)

- Chromium content 3.6 % = 36 000 ppm
- Soluble chromium-(VI) content = 3 ppm, hydrochloric acid extract 0.1 M according to AP(89)1
- Addition of pigment to intermediate (masterbatch) = 5 %
- Addition of intermediate (masterbatch) into the FCM = 6 %

Calculation with soluble content:

→ 3 ppm x 5 % = 0.15 ppm
→ 0.15 ppm x 6 % = **0.009 ppm** [chromium (VI) content in FCM, 100 % migration is unrealistic]

Calculation according to the EFSA tool in "*Note for guidance for petitioners presenting an application for the safety assessment of a substance to be used in food contact materials prior to its authorization*", point 3. Calculation of the maximum possible migration:

$$M = \frac{Q \times A \times Lp \times D}{1000}$$

Variable description:

- M = the **maximum possible migration** of the substance, expressed in mg/kg food
- Q = Calculated amount of the substance based on an analysis (hydrochloric acid extract) in mg/kg Polymer (FCM), from our calculation example, Pigment Brown 24, inorganic coloured pigment = **0.009 mg/kg chromium-(VI) in the FCM**
- A = the area of the food contact material in cm^2, which is usually set at **600 cm^2**
- Lp = the thickness of food contact materials in cm. The maximum thickness can be set at **0.025 cm**, a maximum migration is conventionally expected
- D = the density of the polymer in g/cm^3, here **1.0 g/cm^3** as an example

Calculation example:

$$M = \frac{Q \times A \times Lp \times D}{1000} = \frac{0{,}009\ mg/kg \times 600\ cm^2 \times 0{,}025\ cm \times 1{,}0\ g/cm^3}{1000} = 0{,}000135\ mg/kg$$

0.000135 mg/kg is the maximum migration of chromium-(VI) to be expected from the inorganic coloured pigment (pigment Brown 24) in the FCM, which is considerably below the tolerable level for chromium-(VI), < 0.0012 mg/kg, recital (19) of Regulation (EU) 2020/1245.

When considering the total chromium SML, due to this pigment, the SML should be 3.6 mg/kg instead of 0.01 mg/kg.

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The Verband der Mineralfarbenindustrie e. V. represents German manufacturers of inorganic (e. g. titanium dioxide, iron oxides), organic and metallic pigments, fillers (e. g. silica), carbon black, ceramic and glass colours, food colourants, artists' and school paints, masterbatches and products for applied photocatalysis.

The VdMi is listed in the Lobbying Register for the Representation of Special Interests vis-à vis the German Bundestag and the Federal Government (Lobbyregister des Deutschen Bundestags, number R000760) as well as in the Transparency Register of the EU Commission (number 388728111714-79).